

BEAMLINE X7A

PUBLICATION

A. Burton, S. Elomari, R. Medrud, I. Chan, C. Chen, L. Bull, E. Vittoratos, "The Synthesis, Characterization, and Structure Solution of SSZ-58: A Novel Two-Dimensional 10-Ring Pore Zeolite with Previously Unseen Double 5-Ring Subunits," *J. Am. Chem. Soc.*, **125**, 1633-1642. (2003)

FUNDING

ChevronTexaco Strategic Research

FOR MORE INFORMATION

Allen Burton, Chevron Research & Technology Company
buaw@chevrontexaco.com

The Structure Solution of SSZ-58: a Novel Two-Dimensional 10-Member-Ring-Pore Zeolite with Previously Unseen Double Five-Member-Ring Subunits

Allen Burton, Saleh Elomari, Ronald C. Medrud, Ignatius Y. Chan, Cong-Yan Chen, Lucy M. Bull, and E. Steven Vittoratos

ChevronTexaco

Researchers at ChevronTexaco have recently prepared a novel zeolite (designated SSZ-58) with a ten-dimensional system of ten-membered ring pores. SSZ-58 is the first zeolite to possess a previously unobserved double five-membered ring unit. The structure of SSZ-58 was determined using the ab initio method FOCUS with powder diffraction data collected on beamline X7A. To date, SSZ-58 is the most complex zeolite (in terms of the number of atoms in the crystallographic asymmetric unit) to be solved from powder diffraction data.

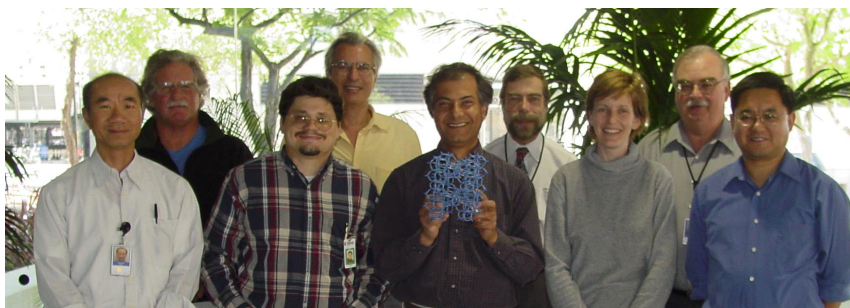
Zeolites are crystalline microporous aluminosilicates which find widespread use as catalysts, adsorbents, and ion-exchangers. New zeolites continue to emerge at an increasingly rapid pace. The syntheses of high-silica zeolites are usually performed hydrothermally in the presence of an organocationic structure directing agent (SDA). The size and shape of the SDA often correlate well with the dimensions of the zeolite micropores or cages. An understanding of molecular recognition effects in zeolite synthesis begins with identification of the structure or structures promoted by certain SDAs. A detailed knowledge of zeolite structure is also crucial for understanding catalytic and adsorptive properties. Once the structure of a zeolite is determined, applications based upon size or shape-selective processes may be targeted, or known properties of the material may be rationalized. Although structure solutions of zeolites from single crystals have been reported, it is rare that zeolite crystals are large enough for single crystal structure analyses of novel zeolite phases. Investigators must therefore rely on powder diffraction data. *Ab initio* structure solution from powder data is difficult because of the high degree of peak

overlap inherent to most powder patterns.

In the past, there was often a long time between the initial discovery of a zeolite and its structure elucidation. The investigators hoped to construct a model that was consistent with the unit cell parameters, the possible space group symmetries, and other physicochemical (*i.e.*, adsorption, catalytic, and density) data. These structure solutions often depended on simple modifications of previously known zeolite topologies. Model building continues to be an important tool to the zeolite crystallographer, particularly for the investigation of disordered materials. However, if an

unsolved structure contains previously unobserved secondary building units and especially if it possesses a large number of tetrahedral (T) atoms in its asymmetric unit, then the success of model building is more dubious.

Recently with the advent of the FOCUS[®] and ZEFSAII algorithms, there have been significant advances in *ab initio* structure solution of zeolites from powder diffraction data. These methods incorporate the crystal chemical information inherent to most zeotype structures: *i.e.*, a fully connected, tetrahedral arrangement of the dominant x-ray scatterers. In this report we discuss the structure so-



Authors (from left to right in front row): Ignatius Chan, Allen Burton, Saleh Elomari, Lucy Bull, Cong-Yan Chen. (From left to right in back row): Stacey Zones, Steven Vittoratos, Charles Wilson (Catalyst Group Manager), Charles Kibby. Ronald Medrud is not shown (he's now retired). Saleh Elomari is holding a model of SSZ-58, but it appears too small.

lution of the novel zeolite SSZ-58 by the FOCUS method.

SSZ-58 was synthesized using the 1-butyl-1-cyclooctylpyrrolidinium cation as a structure directing agent (SDA). Samples for detailed structural analysis were examined at Beamline X7A at Brookhaven National Laboratory. The synchrotron powder diffraction pattern (**Figure 1**) of SSZ-58 could be indexed in an orthorhombic unit cell in space group *Pmma*: $a = 25.112 \text{ \AA}$, $b = 12.498 \text{ \AA}$, $c = 12.860 \text{ \AA}$. **Figure 2** shows the topological structure of SSZ-58 (oxygen atoms have been omitted for clarity) determined from the FOCUS algorithm. This structure has 12 tetrahedral (T) at-

oms and 26 oxygen atoms per asymmetric unit. In terms of the number of unique T atoms, SSZ-58 is the most complex zeolite or zeotype structure solved from powder diffraction data.

SSZ-58 possesses a two-dimensional system of pores that intersect to form large cavities that are bound by two pairs of opposing 10-membered rings (10MRs). The pores along the *c*-direction are straight channels bound by 10-MRs with dimensions of $5.7 \times 5.2 \text{ \AA}$ assuming an oxygen radius of 1.35 \AA . The sinusoidal pores along the *a*-direction are bound by distorted elliptical 10MRs with dimensions of $4.8 \times 5.7 \text{ \AA}$. The structure of SSZ-

58 is composed of columns that possess double five-member rings (D5MRs). Although the D5MR is a simple building unit, it surprisingly has not been observed in any other zeolite structures. The structure solution of SSZ-58 highlights the advances that have been made in structure elucidation of complex materials from powder data.

Works Cited:

1. Breck, D. W. *Zeolite Molecular Sieves: Structure, Chemistry, and Use*, Wiley: New York, 1974
2. Baerlocher, C.; Meier, W. M.; Olson, D. H. *Atlas of Zeolite Framework Types*, 5th ed; Elsevier: New York 2001. Also available at www.iza-structure.org
3. Lobo, R. F.; Zones, S. I.; Davis, M. E. *J. Inclusion Phenom. Molec. Recognition Chem.* **1995**, *21*, 47.
4. Chen, C. Y.; Finger, L. W.; Medrud, R. C.; Crozier, P. A.; Chan, I. Y.; Harris, T. V.; Zones, S. I. *Chem. Commun.* **1997**, 1775.
5. Cambor, M. A.; Diaz-Cabanas, M. J. Perez-Pariente, J.; Teat, S. J.; Clegg, W.; Shannon, I. J.; Lightfoot, P.; Wright, P. A.; Morris, R. E. *Angew. Chem., Int. Ed.* **1998**, *37*, 2122.
6. Grosse-Kunstleve, R. W.; McCusker, L. B.; Baerlocher, C. *J. Appl. Cryst.* **1997**, *30*, 985.
7. Grosse-Kunstleve, R. W. Dissertation ETH No. 11422, 1996.
8. Falcioni, M.; Deem, M. W. *J. Chem. Phys.* **1999**, *110*, 1754.

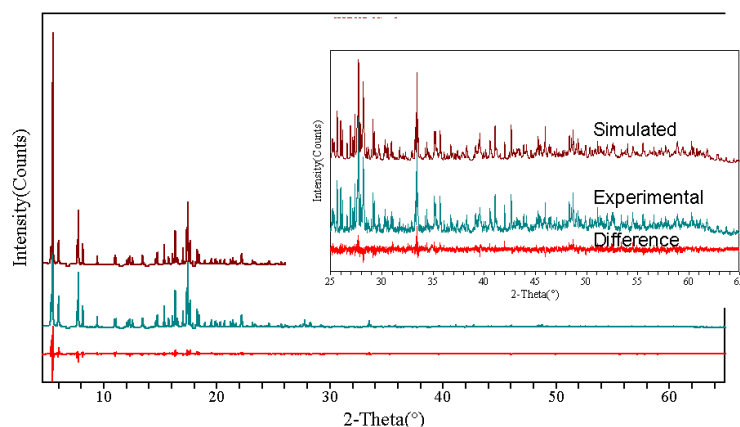


Figure 1. The simulated, experimental, and difference profiles of the synchrotron powder x-ray diffraction pattern ($\lambda = 1.1996 \text{ \AA}$) of SSZ-58.

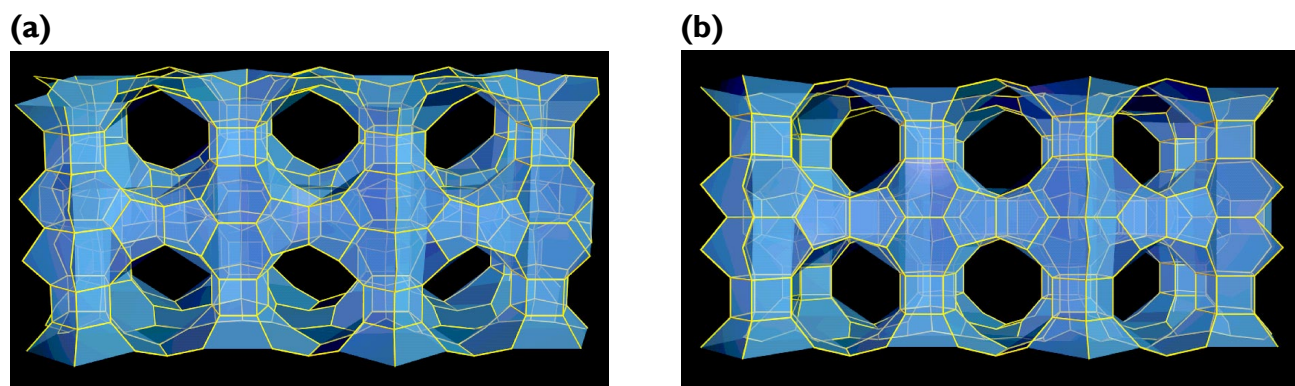


Figure 2. Models showing the two-dimensional pore system in SSZ-58 (a) along the *c*-axis and (b) along the *a*-axis. Tetrahedral atoms are represented by intersections of line segments. Oxygen atoms have been removed for visual clarity.